NASA TM X 50852

31 p.

N63 23040

CODE-1

(MTP-P&VE-M-63-9)

July 16, 1963 3 ref

GEORGE C. MARSHALL SPACE FLIGHT CENTER

HUNTSVILLE, ALA.

(NASA TM X 50852;

APPARATUS FOR DETERMINING LOW TEMPERATURE THERMAL CONDUCTIVITY

By

[6]

C. F. Smith

OTS PRICE

XEROX $ 3.60 ph

MICROFILM $ 1.13 mf.

[redacted]

AERONAUTICS AND SPACE ADMINISTRATION

MSFC - Form 523 (Rev. November 1960)

GEORGE C. MARSHALL SPACE FLIGHT CENTER

MTP-P&VE-M-63-9

APPARATUS FOR DETERMINING LOW TEMPERATURE THERMAL CONDUCTIVITY

By

C. F. Smith

ABSTRACT

23040

Because of the need for low temperature thermal conductivity data on a wide variety of materials used in the space launch vehicle field, an apparatus was developed for determining such information on metal over a temperature range of 80° to 225°K (-316° to -55°F). Data are presented for copper, stainless steel, and aluminum alloys and compare quite favorably with values reported by other investigators on the same alloys.

AUTHOR

GEORGE C. MARSHALL SPACE FLIGHT CENTER

MTP-P&VE-M-63-9

APPARATUS FOR DETERMINING LOW TEMPERATURE THERMAL CONDUCTIVITY

By

C. F. Smith

ENGINEERING MATERIALS BRANCH
PROPULSION AND VEHICLE ENGINEERING DIVISION

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

GEORGE C. MARSHALL SPACE FLIGHT CENTER

MTP-P&VE-M-63-9

APPARATUS FOR DETERMINING LOW TEMPERATURE THERMAL CONDUCTIVITY

By

C. F. Smith

SUMMARY

Because of the need for low temperature thermal conductivity data on a wide variety of materials used in the space launch vehicle field, an apparatus was developed for determining such information on metal over a temperature range of 80° to 225°K (-316° to -55°F). Data were collected on copper, stainless steel, and aluminum alloys. Accuracy of the data was within 10 percent of values reported by other investigators on the same alloys. This was considered good agreement in view of the normal variation in composition of commercial alloys and since no absolute values or standard test samples have been established for comparison of thermal conductivity data between different investigators.

INTRODUCTION

Determining the thermal conductivity of materials at low temperature is necessary for the advancement of many scientific fields. This is particularly true in the space launch vehicle field where cryogenic propellants are used extensively. As new materials are developed for more exacting uses at temperature extremes, the inherent properties of these materials must be determined before they can be utilized fully. Thermal conductivity data in the cryogenic range for many of these materials are not readily available, if at all.

The purpose of this paper is to describe an apparatus which was developed for measuring the thermal conductivity of specific materials at cryogenic temperatures. Since the type material and exact temperature range over which the conductivity is to be determined cannot always be anticipated prior to need, it was most desirous to have an apparatus with wide parameters. This being the case, a steady state method of axial heat flow through a long cylindrical sample was employed since it would yield the most data for a given thermal equilibrium.

Acknowledgement

The author is indebted to Leo D. Wilson, Guidance Systems Section, Guidance and Controls Systems Branch, Astrionics Division, for designing the zener diode regulated power supplies used in this program and to Wayman N. Clotfelter and Luke A. Soileau, Engineering Materials Branch, Propulsion and Vehicle Engineering Division, for their assistance in setting up the equipment.

DESIGN AND CONSTRUCTION OF APPARATUS

General Description

A diagram of the apparatus is shown in FIG 1. FIG 2 is a view of the associated measuring equipment. This apparatus is a modification of the one developed by Powell, Rogers, and Coffin (Ref. 1). A long cylindrical specimen is clamped at one end to a heavy copper plate, which acts as a heat sink. The heat sink is immersed in a cryogenic fluid, in this case, liquid nitrogen. The other end of the specimen is heated by a suitably guarded heater, and the temperature gradient across the sample is measured by eight equally spaced thermocouples. To prevent heat losses, a copper radiation shield is clamped over the specimen heater. This is then enclosed in another radiation shield which is attached to the heat sink. The temperature gradient of this outer shield is regulated by three guard heaters which are wound on the radiation shield. The lower heater is regulated by a servo mechanism to maintain the same temperature as the specimen heater. The two other heaters are controlled manually to the approximate temperature of the specimen at the mid-point of the area in which each heater is located.

To minimize heat losses through gas convection and conduction, the entire system is enclosed in a vacuum tight chamber, maintained at a pressure of approximately 10^{-6} mm Hg. The vacuum chamber is, in turn, immersed in a stainless steel dewar filled with liquid nitrogen.

Specimen Arrangement

The metal specimens used in this apparatus were machined to 0.144 ± 0.0005 inch diameter by 10-1/8 inches long. One end was clamped to a copper heat sink by a split bolt clamping arrangement. Copper thermocouple holders, fitted with teflon strips to electrically insulate the measuring junction, were used to attach eight thermocouples to the specimen. The thermocouple holders were assembled on the sample by use of a special gauge block so that the measuring junctions were one-inch apart. Details of the specimen assembly and thermocouple holder are shown in FIG 3.

The thermocouple wire used to measure the temperature gradient along the specimen was #36 gauge copper-constantan (special limits). All thermocouple junctions were made by arcing on a carbon electrode. The reference junction was insulated with fiberglass tape and clamped in one of five copper studs, which were part of the heat sink. A National Bureau of Standards calibrated platinum resistance thermometer was used to determine the reference junction temperature. It was mounted in another copper stud on the heat sink.

The specimen heater was attached to the specimen with a set screw, and consisted of a copper cylinder insulated with General Electric Company #7031 varnish and wound with #40 AWG Alloy "45,"* 48 ohms/ft wire. Its resistance was approximately 60 ohms. The winding was anchored to the copper core by a final coating of General Electric Company #7031 varnish. Thermal contact with the specimen was improved by placing a drop of mercury in the specimen heater prior to inserting the specimen.

All thermocouple and heater wires were wound on the remaining copper studs on the heat sink before leaving the vacuum chamber through Conax** fittings to the measuring equipment. This technique helped to minimize a heat leak by conduction through the leads.

Radiation Shields and Guard Heaters

This apparatus employed three radiation shields and three guard heaters, all of which functioned to prevent a loss or gain of thermal energy in relation to the heated specimen. The innermost radiation shield consisted of a gold plated copper cylinder which encompassed the specimen heater without making physical contact. This shield was attached to the specimen by clamping to a teflon disc, located above the heater. The disc thermally insulated the specimen from the shield and physically supported the shield. The specimen assembly and inner radiation shield were enclosed by a second radiation shield which extended the full length of the specimen assembly. The shield was fabricated from stainless steel and gold plated copper. It was divided in half longitudinally, and the front half was removable for easy access to the specimen assembly. This shield and the entire specimen assembly were enclosed by a stainless steel vacuum cylinder which served as another radiation shield. FIG 4 shows the specimen assembly and guard heaters with the outer radiation shields removed.

The three guard heaters were wound in two sections each on the long cylindrical radiation shield. The heaters were wound in this manner to simplify removal of the shield without requiring the heaters to be rewound with each change of sample. The guard heaters were approximately 325 ohms each. The same type of wire was used in winding these heaters as in the specimen heater. A wiring diagram of the heater arrangement is shown in FIG 5a.

* Trade name of C. O. Jelliff Company

** Trade name of Conax Corporation

The temperature difference between the specimen and guard heaters was measured by differential thermocouples. In the case of the bottom guard heater, the output of the differential thermocouple was fed into a null balance amplifier. This amplifier controlled a motor driven rheostat, which automatically established adiabatic conditions between the bottom guard heater and sample heater. These thermocouples were also assembled from #36 AWG copper-constantan wire. FIG 5b shows a block diagram of the servo system.

Power Supplies and Measuring Equipment

FIG 5c shows a schematic diagram of the specimen heater and power measuring circuits. The power input to the specimen heater was measured across calibrated NBS resistors with a K-3 Leeds and Northrup potentiometer. The total current passed through a one-ohm resistor, and the voltage drop across it was measured. The specimen heater was shunted by a voltage divider consisting of three resistors in series; two 10K ohm, and one 1K ohm resistors. This arrangement gave a voltage divider ratio of 21 to 1. The voltage drop across the 1K ohm resistor was measured and multiplied by 21 to obtain the applied voltage. All resistors were mounted in a thermostatically controlled constant temperature oil bath which was maintained at 26°C (79°F).

A six dial Minneapolis-Honeywell potentiometer was used for measuring the output of the thermocouples used to determine the temperature gradient along the sample. The resistance of the NBS calibrated platinum resistance thermometer used for determining the reference temperature was measured on a five-dial Mueller bridge. The thermometer was calibrated from 4.2° to 100°K (-452° to -280°F) in one degree intervals. Null balance electronic galvanometers were used as detectors with both potentiometers and the Mueller bridge. FIG 5d shows a schematic diagram of the temperature measuring circuits.

Only direct current power supplies of the solid state type, regulated with zener diodes, were used. This type of power source has proven to be very stable. There was an estimated 0.0001 percent change for a 10 percent fluctuation in line voltage. The use of direct current in the power circuits precluded any difficulties in the measuring circuits of a.c. pickup from the heater circuits. FIG 6 shows a basic schematic diagram of the zener diode type regulated power supply used in this apparatus. This type power supply also supplied all battery working sources for the measuring instruments.

Vacuum System and Other Accessory Equipment

A mechanical roughing pump, 425 liters/minute free air capacity, and 4 inch oil diffusion pump were used to maintain a pressure of less than 5×10^{-5} mm Hg. The vacuum was monitored with a thermocouple and an ionization gauge. The cover for the liquid nitrogen dewar was an integral part of the vacuum system. It was composed of an inner and outer vacuum chamber with a liquid nitrogen chamber between the two. The two vacuum chambers were tied directly to the remainder of the vacuum system. An automatic liquid level control apparatus was used to maintain the liquid nitrogen level at approximately two-inches above the heat sink. The liquid nitrogen dewar surrounding the specimen chamber was filled from a pressurized 175 liter reservoir.

DISCUSSION OF PROBLEMS AND SPECIAL TECHNIQUES

In the design and construction of scientific equipment of this type, many unforeseen problems arise which must be surmounted prior to obtaining the desired data. Initially, much difficulty was encountered in attaching the vacuum chamber to the heat sink. Attempts were first made to seal the chamber with Rose's alloy. In using this alloy, some sealing operations would hold a vacuum at room temperature; but, when subjected to liquid nitrogen temperature, the seal would rupture. The ruptured seal would then close on returning to room temperature. Cerrobend was tried with only slightly better results until the technique of preparing the two mating surfaces was changed. The knife edge on the heat sink and trough on the chamber were cleaned with Green Streak Flux* and tinned with 50-50 lead-tin solder. Molten cerrobend was then poured into the preheated trough. The tinned knife edge was also pretreated, and the flux and oxides wiped from the surface before it was immersed in the cerrobend filled trough. A stainless steel tool was worked around in the molten cerrobend to remove any trapped air or oxides that had formed. On cooling, this technique produced a high vacuum seal that would withstand thermal cycling from 20° to -196°C (68° to -321°F).

In the initial testing of this apparatus, high capacity nickel-cadmium batteries were used as a power source for the heaters. These lacked the stability that was expected, considering the small power drain. The absence of stability in this power source made the attainment of thermal equilibrium impossible. The batteries were replaced by the zener regulated power supplies discussed earlier. These units have proven to be extremely stable and maintenance free.

As mentioned previously, the bottom guard heater was controlled by a motor driven rheostat from a null balance amplifier. As far as maintaining thermal equilibrium was concerned, this system was adequate. The main limitation or drawback was that a very close approximation to

* Trade name of Metal Mending Products, Inc.

thermal equilibrium had to be obtained manually before the servo mechanism could be energized. Establishing a close approach to equilibrium in this manner was very time consuming. It is hoped to replace this control system with a three mode type anticipating control unit in the near future, which should aid considerably in increasing the data output.

One other time consuming operation that was shortened was the installation of the specimen. After the specimen and thermocouple holders were clamped in position, the thermocouples were attached to the thermocouple holders with an epoxy potting compound which necessitated making new junctions for each new specimen. The radiation shield surrounding the specimen also made it necessary to wind three guard heaters each time a specimen was installed. These operations were shortened by clamping the thermocouple junction between two teflon strips on the thermocouple holder. In this way, the thermocouple junctions could be readily attached and used again. In addition, halves of each of the three guard heaters were attached to each half of the radiation shield. When the radiation shield was assembled, it was only necessary to connect half of each heater in series with the other half to complete the assembly.

Since the electrical signals to be measured in this apparatus are of very low output, special precautions were taken in wiring the electronic circuits to prevent pickup of extraneous signals from associated equipment, from unrelated equipment in the surrounding area, and from static fields generated by clothing. The measuring equipment was connected with coaxial cable and placed on an insulated sheet of copper, and a common ground used. All thermocouple wire was one continuous piece from the measuring junction inside the vacuum sample chamber to a shielded terminal strip mounted outside the apparatus. These wires extended through vacuum tight Conax fittings equipped with teflon seals. This technique reduced the possibility of stray thermal emfs being developed.

CALCULATIONS

The calculations for determining thermal conductivities from this apparatus are based on the fundamental equation of unidirectional heat flow:

$$Q = -kA \frac{dt}{dx} \tag{1}$$

where Q is the quantity of heat, k the thermal conductivity, A the cross sectional area and dt/dx the temperature gradient. The minus sign is introduced so that the positive direction of heat flow should coincide with the positive direction of x. For heat to flow in the positive direction of x, this must be the direction in which t decreases. Rewriting equation (1)

$$Q = kA \frac{t_1 - t_2}{x_1 - x_2} , \tag{2}$$

where t_1 is the temperature at point x_1 and t_2 the temperature at x_2 on the specimen. No correction was made for changes in A and $x_1 - x_2$ at cryogenic temperatures since these changes are insignificant in comparison to the error in the thermocouple measurements. Prior to taking thermocouple readings for calculating the temperature differential, establishment of equilibrium was verified by taking readings until there was less than one microvolt change for each thermocouple over a period of one hour. The emfs developed at these eight junctions were converted to temperature from a table compiled by the National Bureau of Standards.

Q, the heat input, was calculated from the following formula;

$$Q = I \times E \times 21 \tag{3}$$

The current, I, was a direct measurement across a one-ohm resistor and the voltage, E, was measured across one leg of a voltage divider and multiplied by the voltage divider ratio, 21. This calculation gave the heat input to the specimen, with the assumption that adiabatic conditions were established and all heat passed through the specimen.

DISCUSSION OF RESULTS

The results of experimentation to date are shown in Tables I-V. The materials chosen for checking the accuracy and precision of this apparatus exemplify the conductivity ranges covered by the more common metals of construction, with copper having a high conductivity, stainless steel being relatively low, and aluminum being intermediate. FIGS 7-11 compare the data with values reported by other investigators (Ref. 2 and 3). Agreement was within about 10 percent or less of the reported values. This agreement is considered to be fairly close in view of the normal variation in the chemical composition of metal alloys, and the resultant effects on thermal conductivity. In addition, the lack of any standardized thermal conductivity values or standard test specimens prevent an exact comparison of the accuracy of test data between investigators. Consequently, a more rigorous evaluation of errors could not be made.

Through experimentation, it was found necessary to determine an optimum heat flow for each material which establishes the temperature differential. A differential of approximately 4°K (7°F) yielded the best data for copper while a difference of approximately 10° to 15°K (18° to 27°F) was optimum for 2024-T4 aluminum, 10° to 20°K (18° to 36°F) for 7075-T6 aluminum, and 5° to 15°K (9° to 27°F) for 5052-0 aluminum. The optimum temperature differential for 304 stainless steel was comparable to 2024-T4 aluminum.

CONCLUSIONS AND RECOMMENDATIONS

The apparatus described in this report yields good multiple point results over a temperature range of 80° to 225°K (-316° to -55°F) when the heat flow is restricted to the optimum for the range of thermal conductivity being measured. This apparatus can be adapted to accept rigid plastics by modifying the heat sink clamp and specimen heater. The temperature range can be extended down to 4.2°K (-452°F) by using liquid helium as the cryogenic fluid. This will require the use of another dewar and the necessary liquid level control equipment. However, prior to the use of liquid helium, it will be necessary to decrease the time required to reach thermal equilibrium. FIG 12 shows a diagram of the proposed setup for use with liquid helium. As soon as time is available, it is planned that both of these modifications will be made in order to provide a more versatile apparatus applicable to a wider range of measurements.

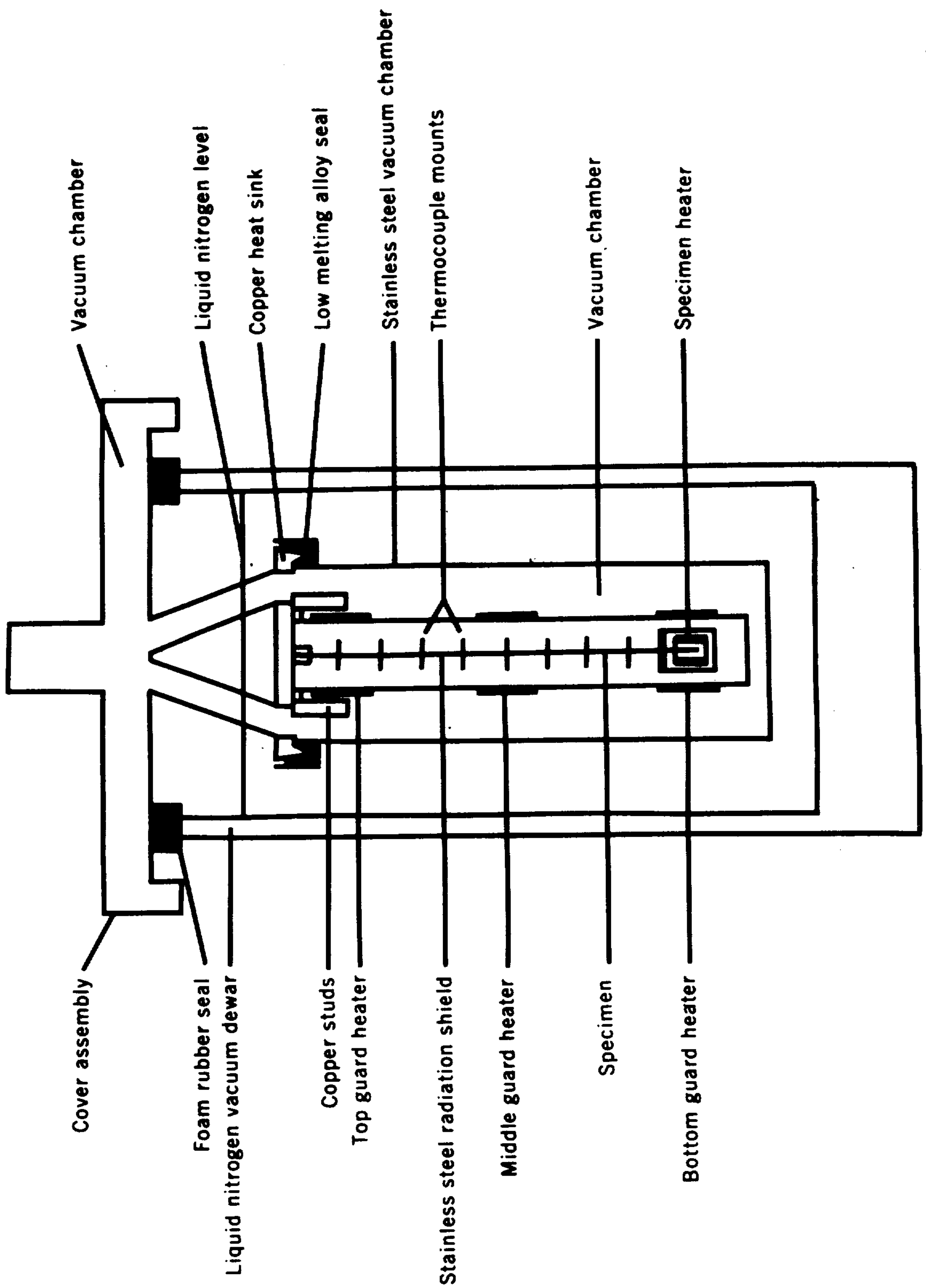


FIGURE 1 DIAGRAM OF THERMAL CONDUCTIVITY APPARATUS

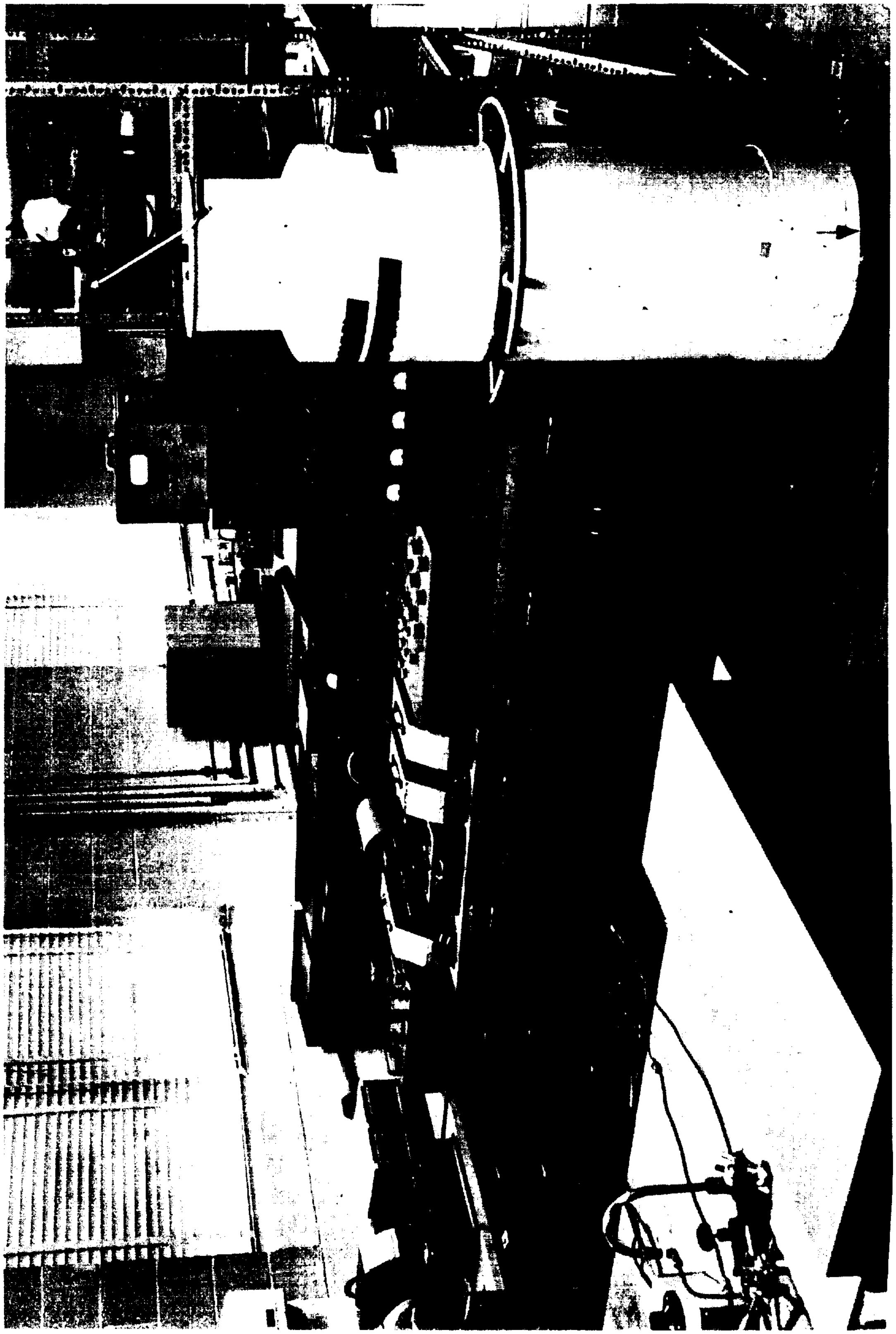
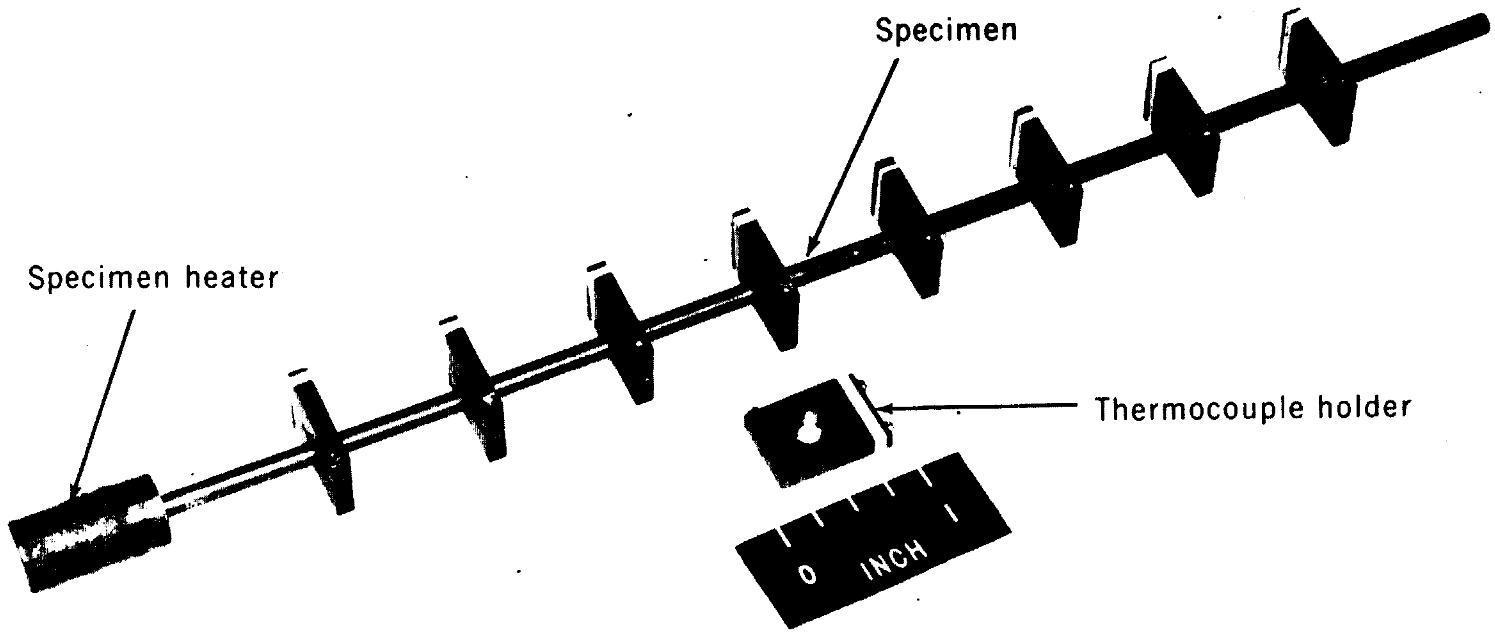


FIGURE 2 THERMAL CONDUCTIVITY APPARATUS AND ASSOCIATED EQUIPMENT



MTP - P&VE - M - 63 - 9

FIGURE 3 DETAILS OF SPECIMEN ASSEMBLY AND THERMOCOUPLE HOLDER

FIGURE 4 RADIATION SHIELDS AND ASSEMBLED SPECIMEN

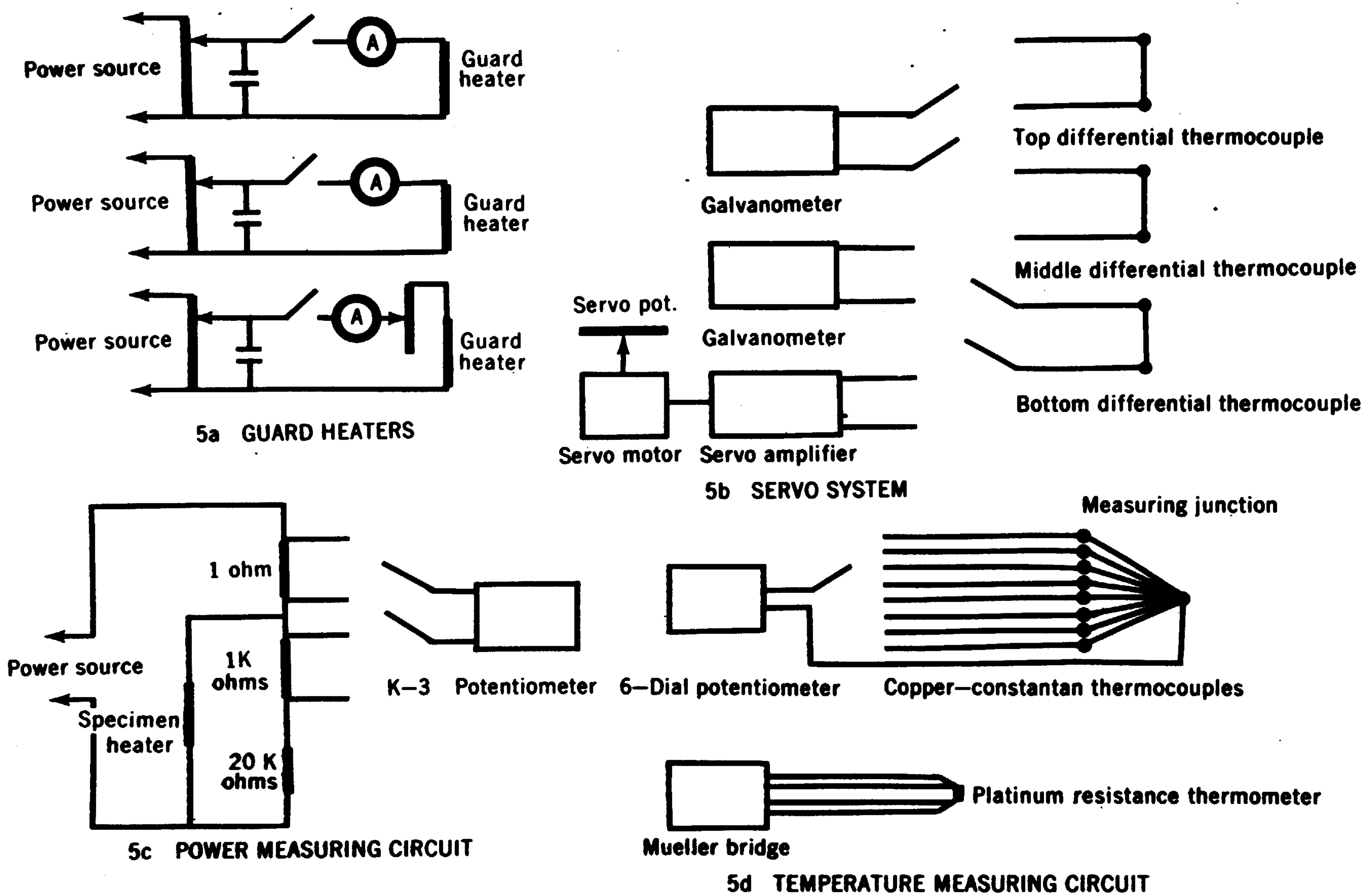


FIGURE 5 ELECTRICAL SCHEMATIC

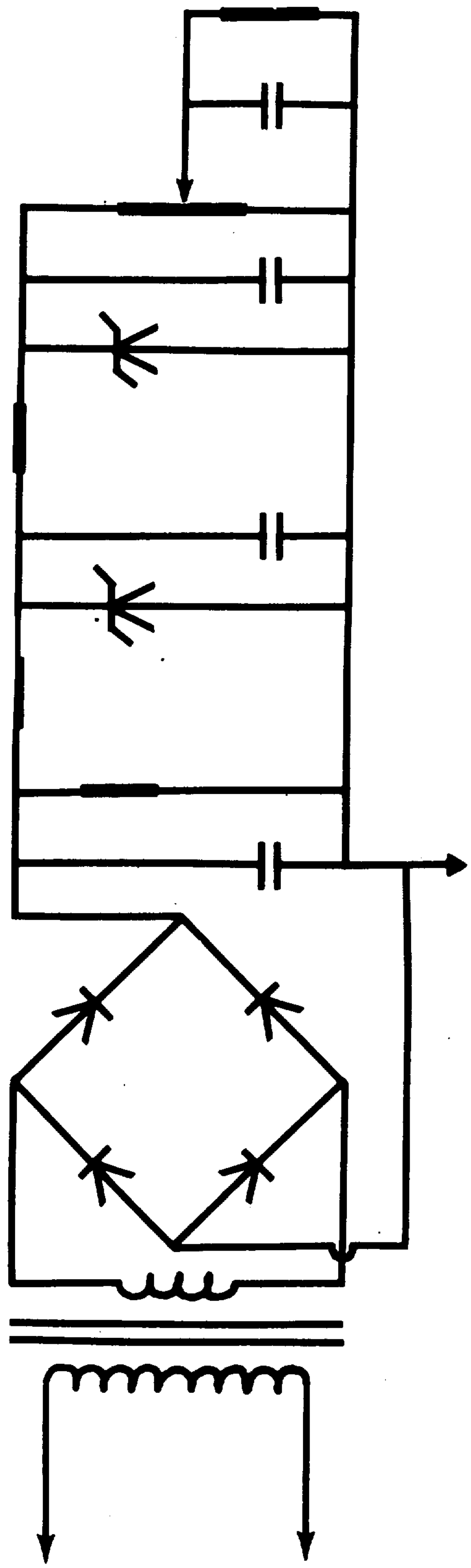


FIGURE 6 BASIC SCHEMATIC OF ZENER DIODE TYPE REGULATED POWER SUPPLY

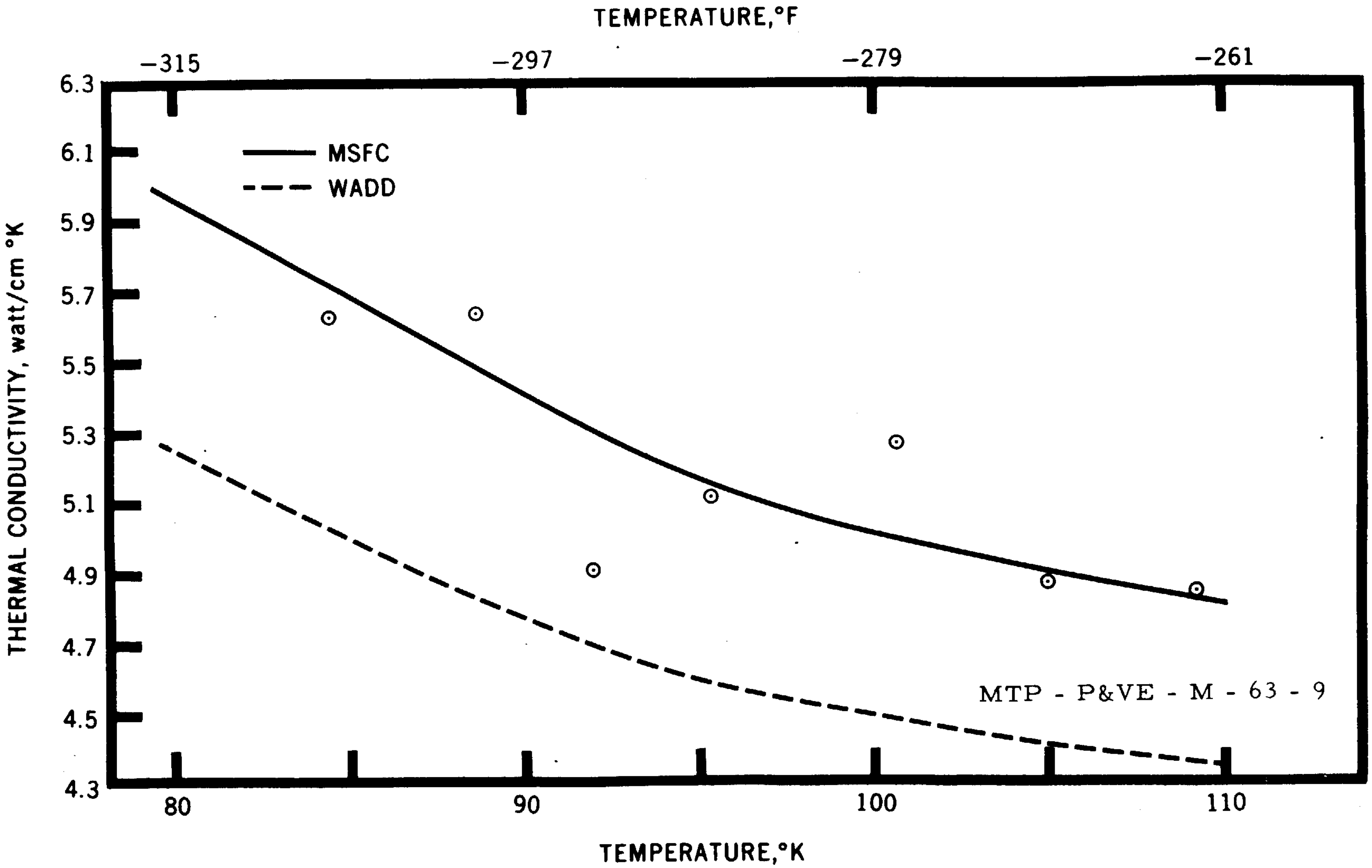


FIGURE 7 THERMAL CONDUCTIVITY OF ELECTROLYTIC COPPER

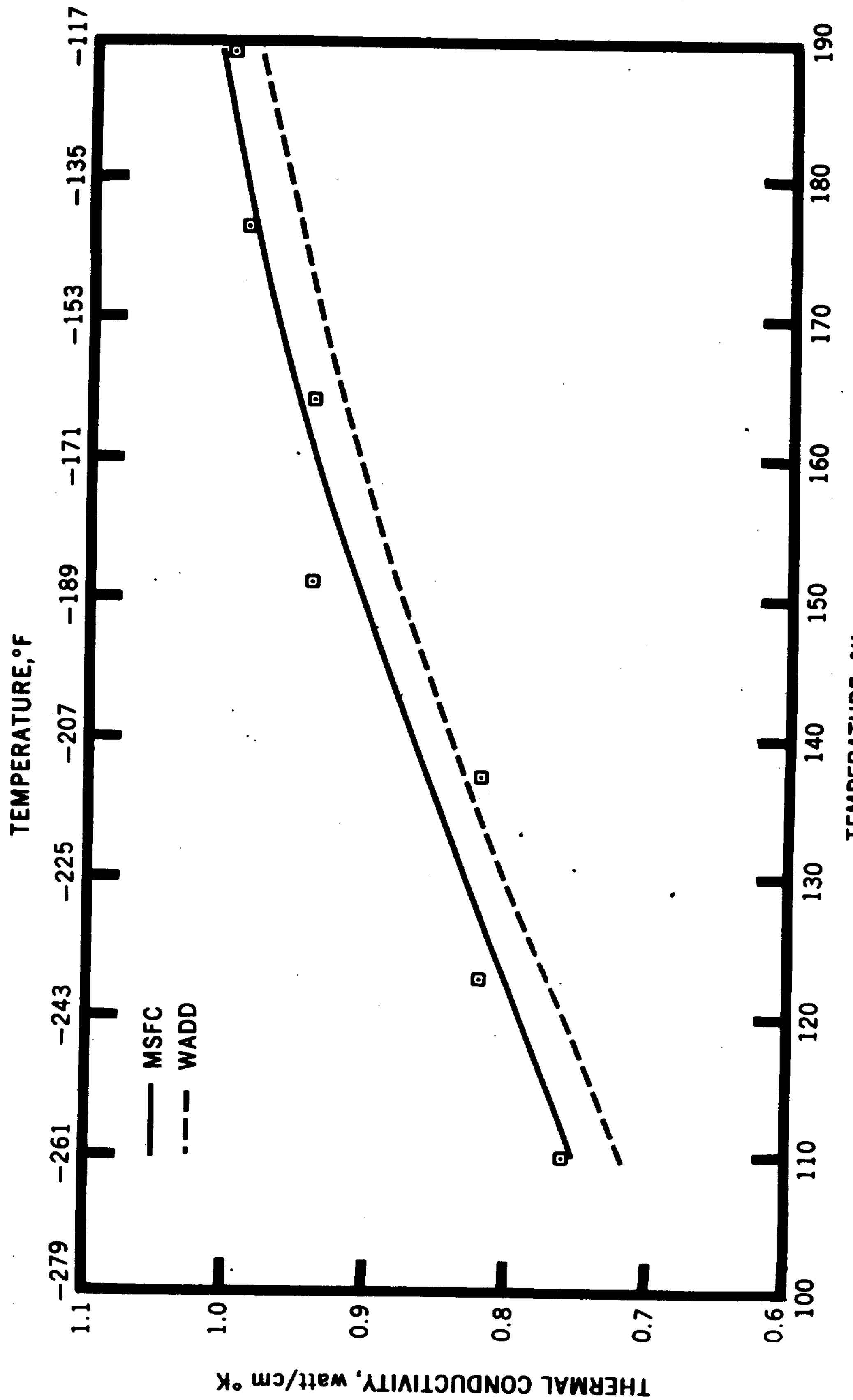


FIGURE 8 THERMAL CONDUCTIVITY OF 2024-T4 ALUMINUM

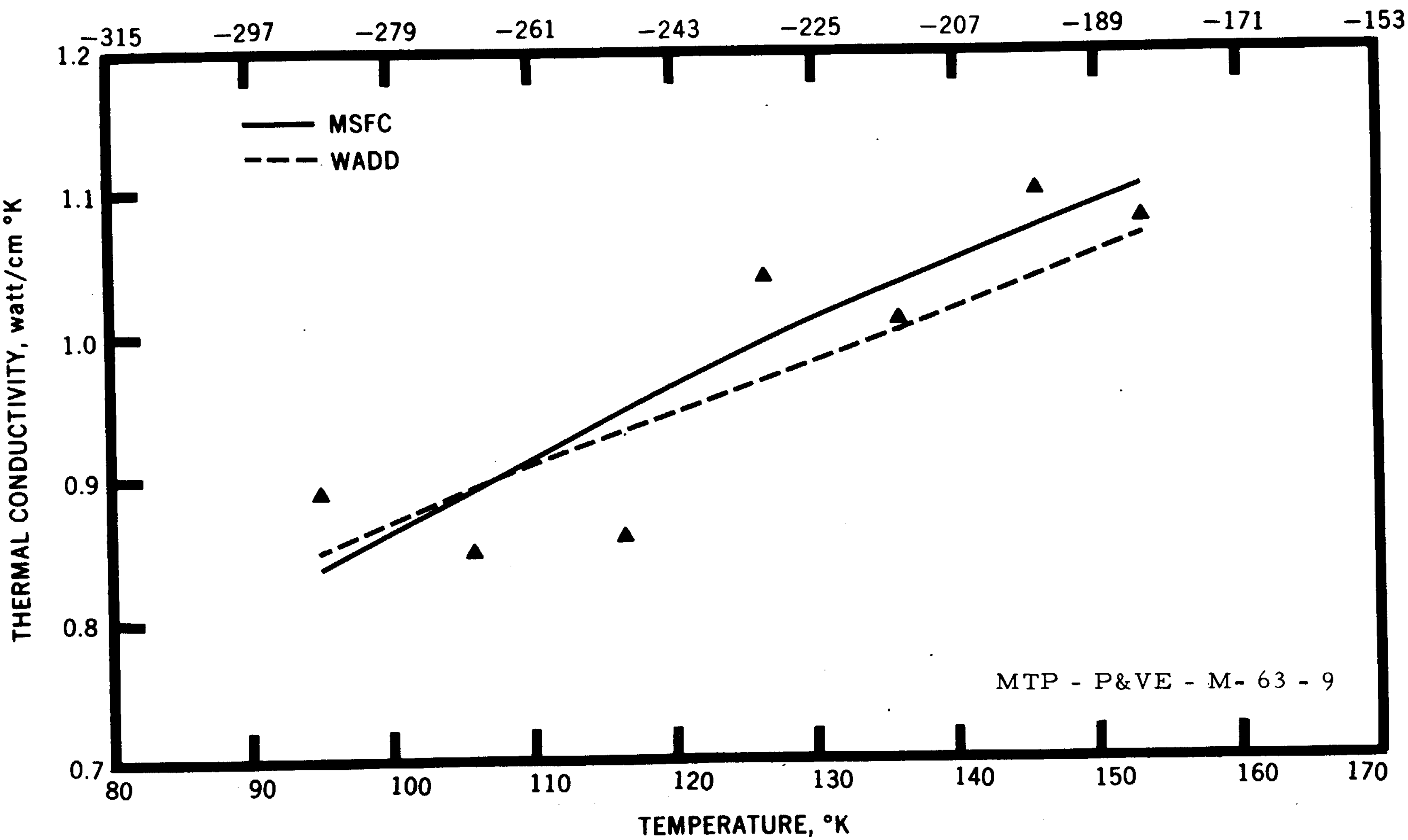


FIGURE 9 THERMAL CONDUCTIVITY OF 5052-0 ALUMINUM

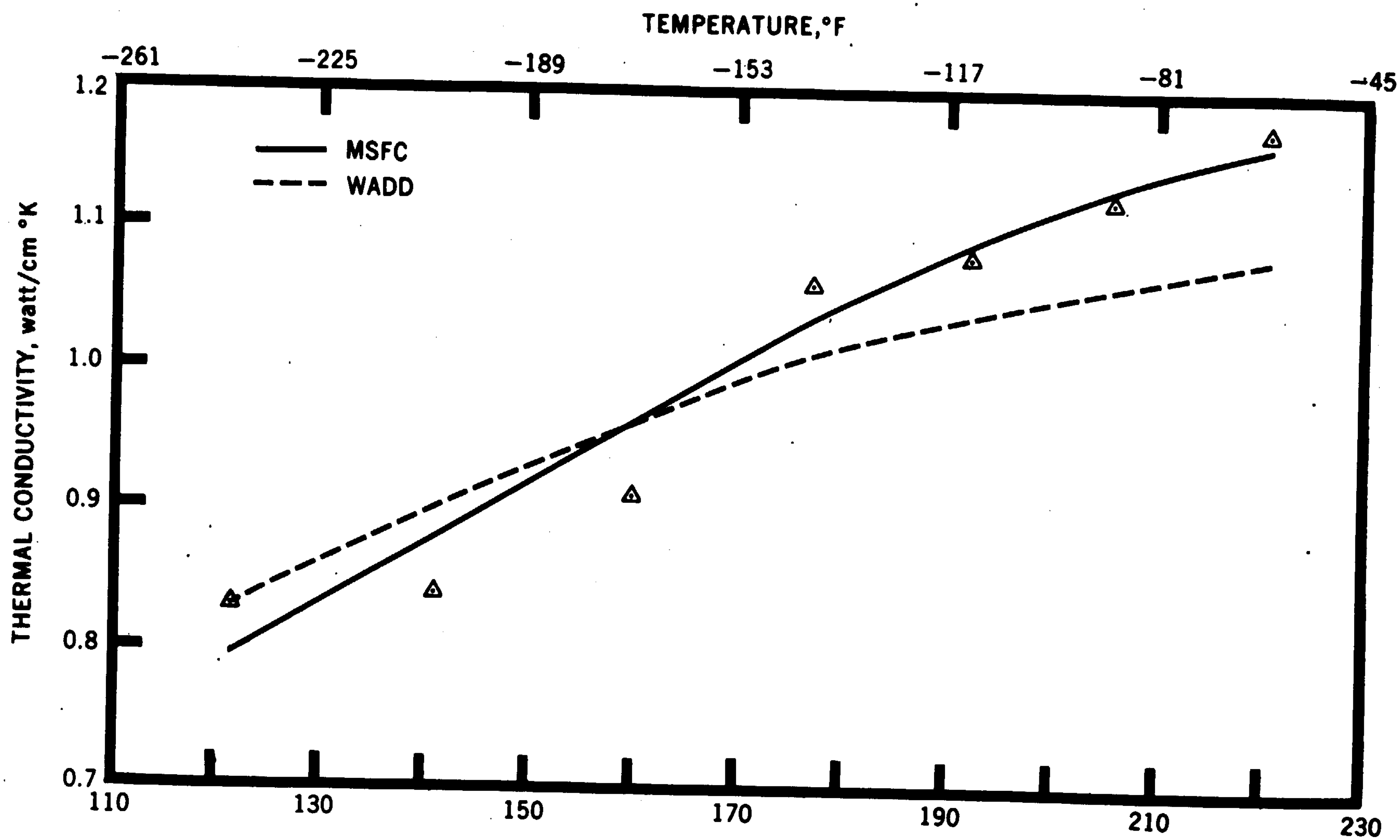


FIGURE 10 THERMAL CONDUCTIVITY OF 7075-T6 ALUMINUM

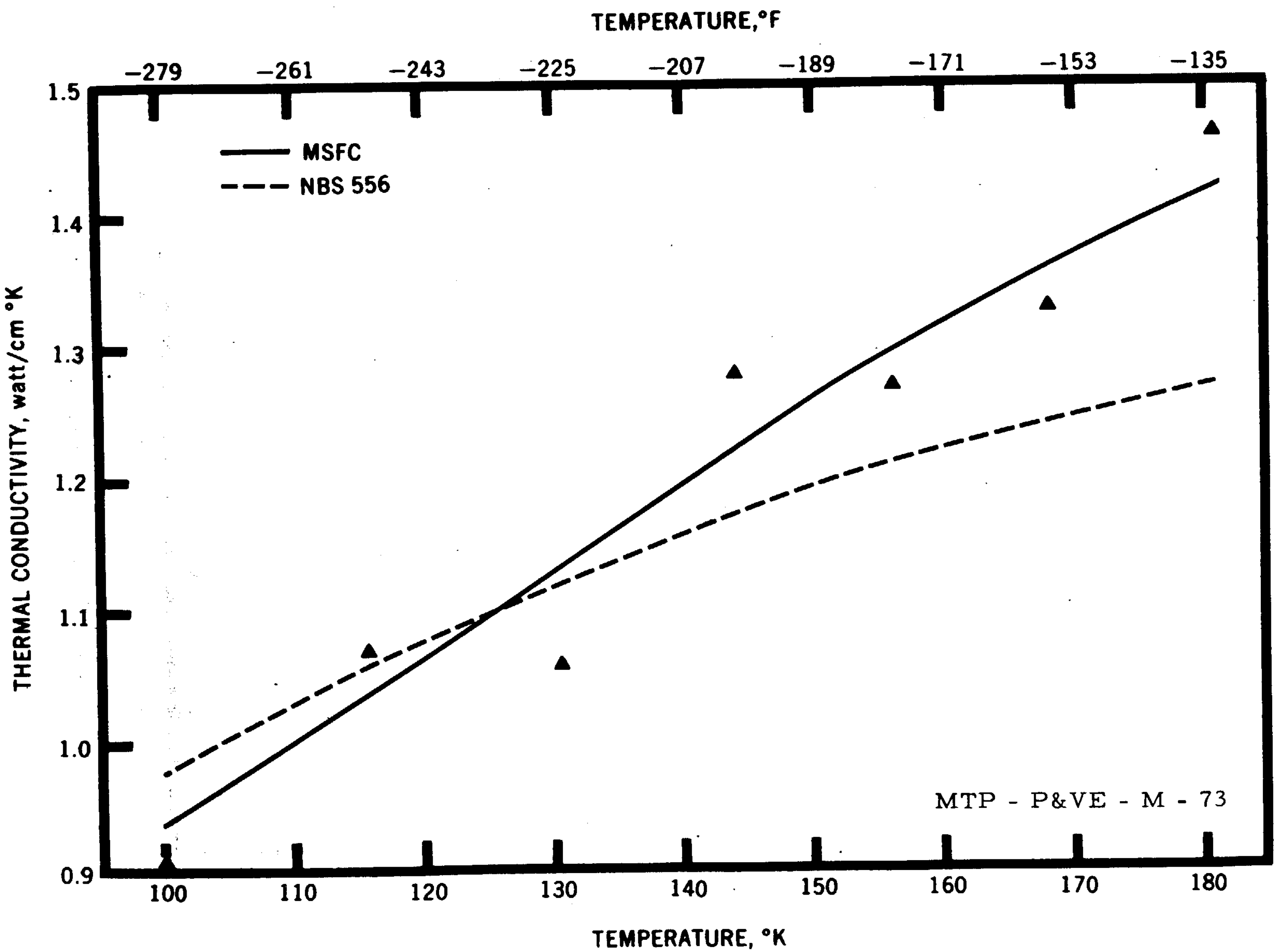


FIGURE 11 THERMAL CONDUCTIVITY OF 304 STAINLESS STEEL

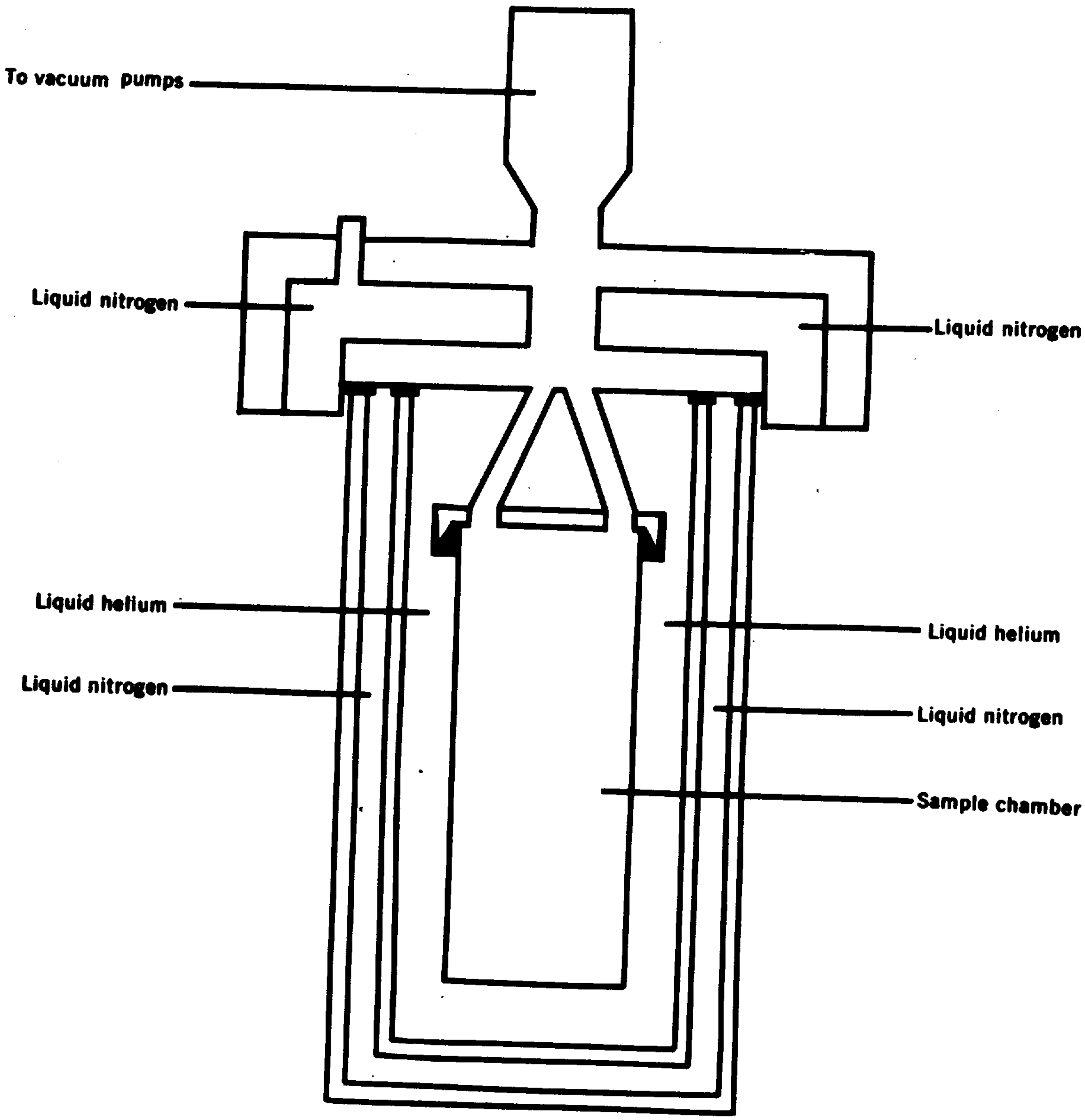


FIGURE 12 PROPOSED APPARATUS FOR USE WITH LIQUID HELIUM

TABLE I

THERMAL CONDUCTIVITY OF ELECTROLYTIC TOUGH PITCH COPPER

Temperature Differential, Δt (°K)	Average Temperature °K	°F	k (watts/cm-°K)
3.78	84	-308	5.63
3.77	88	-301	5.64
4.33	92	-294	4.91
4.15	97	-285	5.12
4.03	101	-278	5.27
4.36	105	-271	4.88
4.38	109	-263	4.86

$Q = 0.876$ Watts
$l = 2.55$ cm
$A = 0.105$ cm^2

TABLE II

THERMAL CONDUCTIVITY OF 2024-T4 ALUMINUM

Temperature Differential, Δt (°K)	Average Temperature °K	°F	k (watts/cm-°K)
16.00	107	-267	0.76
14.76	123	-238	0.82
14.82	137	-213	0.82
12.83	151	-188	0.94
12.86	164	-164	0.94
12.26	177	-141	0.98
12.03	189	-119	1.00

$Q = 0.498$ Watts
$l = 2.55$ cm
$A = 0.105$ cm^2

TABLE III

THERMAL CONDUCTIVITY OF 5052-0 ALUMINUM

Temperature Differential, Δt (°K)	Average Temperature °K	°F	k (watts/cm-°K)
10.48	95	-289	0.89
11.00	106	-269	0.85
10.90	117	-249	0.86
8.97	127	-231	1.04
9.54	136	-215	0.98
8.45	145	-199	1.11
8.59	153	-184	1.09

Q = 0.386 Watts
l = 2.55 cm
A = 0.105 cm^2

TABLE IV

THERMAL CONDUCTIVITY OF 7075-T6 ALUMINUM

Temperature Differential, Δt (°K)	Average Temperature °K	°F	k (watts/cm-°K)
19.41	122	-240	0.83
19.07	141	-204	0.84
18.82	160	-172	0.86
15.12	177	-141	1.06
14.88	192	-114	1.08
14.36	207	- 87	1.12
13.71	221	- 62	1.17

Q = 0.663 Watts
l = 2.55 cm
A = 0.105 cm^2

TABLE V

THERMAL CONDUCTIVITY OF 304 STAINLESS STEEL

Temperature Differential, Δt (°K)	Average Temperature °K	°F	k (watts/cm-°K)
17.14	100	-280	0.090
14.60	116	-251	0.106
14.72	131	-224	0.105
12.14	144	-200	0.128
12.31	156	-179	0.126
11.72	168	-157	0.132
10.69	180	-136	0.145

$Q = 0.638$ Watts
$l = 2.55$ cm
$A = 0.105$ cm^2

REFERENCES

1. Powell, R. L., Rogers, W. M., and Coffin, D. O., "An Apparatus for Measurement of Thermal Conductivity of Solids at Low Temperatures," Journal of Research of the National Bureau of Standards, 59, 5 Nov. 1957, RP 2805.

2. Powell, R. L. and Blanpied, Wm. A., "Thermal Conductivity of Metals and Alloys at Low Temperatures," N.B.S. Circular 556, Sept. 1, 1954.

3. Johnson, Victor J., General Editor, "A Compendium of the Properties of Materials at Low Temperatures (Phase I) Part II. Properties of Solids," Oct. 1960, WADD TR 60-56, Part II.

July 16, 1963 APPROVAL MTP-P&VE-M-63-9

APPARATUS FOR DETERMINING LOW TEMPERATURE THERMAL CONDUCTIVITY

By

C. F. Smith

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

D. B. Franklin

D. B. FRANKLIN
Chief, Corrosion and Physical Chemistry Section

W. R. Lucas

W. R. LUCAS
Chief, Engineering Materials Branch

W. A. Mrazek

W. A. MRAZEK
Director, Propulsion and Vehicle Engineering Division

DISTRIBUTION

M-P&VE-DIR	Dr. Mrazek
M-P&VE-DIR	Mr. Weidner
M-P&VE-E	Mr. Schulze
M-P&VE-F	Mr. Goerner
M-P&VE-M	Dr. Lucas (5)
M-P&VE-MC	Mr. Franklin (10)
M-P&VE-ME	Mr. Kingsbury
M-P&VE-MG	Mr. Riehl
M-P&VE-MM	Mr. Cataldo
M-P&VE-P	Mr. Paul
M-P&VE-PA	Mr. Thompson
M-P&VE-PE	Mr. Bergeler
M-P&VE-PH	Mr. Connell
M-P&VE-PL	Mr. Reed
M-P&VE-PM	Mr. Fuhrmann
M-P&VE-PT	Mr. Wood
M-P&VE-S	Mr. Kroll
M-P&VE-SA	Mr. Blumrich
M-P&VE-SB	Mr. Verble
M-AERO-DIR	Dr. Geissler
M-AERO-A	Mr. Dahm
M-AERO-G	Mr. Vaughan
M-ASTR-DIR	Dr. Haeussermann
M-ASTR-R	Mr. Taylor
M-ASTR-RP	Mr. Reinbolt
M-QUAL-M	Mr. Urbanski (3)
M-RP-DIR	Dr. Stuhlinger
M-RP-A	Mr. Thompson
M-RP-P	Mr. Johnson
M-RP-T	Mr. Heller
M-ME-M	Mr. Wilson
M-MS-IP	Mr. Remer
M-MS-IPL (8)	
M-P&VE-ADMP	Mr. Hofues
M-MS-H	Mr. Akens
M-LGL-P	Mr. Reece
M-PAT	
M-HME-P	

Scientific and Technical Information Facility (2)
ATTN: NASA Representative (S-AK/RKT)
P. O. Box 5700
Bethesda, Maryland